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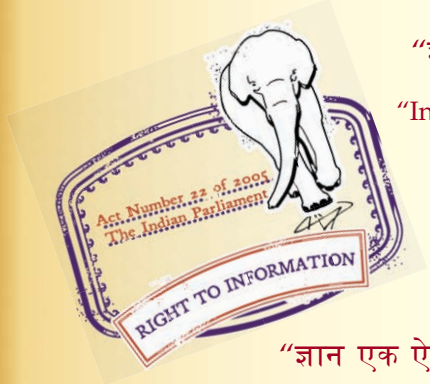

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METHODS OF TEST FOR VULCANIZED RUBBERS

PART XXI DETERMINATION OF PERMEABILITY TO GASES — CONSTANT PRESSURE METHOD

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PART XXI DETERMINATION OF PERMEABILITY TO GASES — CONSTANT PRESSURE METHOD

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Indian Standard

METHODS OF TEST FOR VULCANIZED RUBBERS

PART XXI DETERMINATION OF PERMEABILITY TO GASES — CONSTANT PRESSURE METHOD

0. FOREWORD

0.1 This Indian Standard (Part XXI) was adopted by the Indian Standards Institution on 8 October 1980, after the draft finalized by the Rubber Products Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 The measurement of the permeability of rubber to gases is important in the evaluation of compounds for such products as inner tubes, tubeless tyre liners, hoses, balloons or other gas containers, seals and diaphragms. The measurement is also of theoretical importance in the study of characteristics of diffusion and gas solubility in relation to polymer structure.

0.3 Methods of test for vulcanized rubbers-permeability to gases by constant volume method has already been published as IS : 3400 (Part XIX)-1976.

0.4 Because of dangers connected with high pressure and flammability, handling of gases should be done by experienced personnel only.

0.5 This standard is essentially based on ISO 2782-1977 'Rubber, vulcanized — Determination of permeability to gases — constant pressure method, published by International Organization for Standardization.

0.6 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

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1. SCOPE

1.1 This standard (Part XXI) prescribes the method for determination of permeability of vulcanized rubbers to gases at steady state condition, when a constant pressure is maintained on each of the end faces of the rubber to be tested. Results obtained by this test procedure should not be extrapolated to thickness of material considerably different from that of the test piece.

*Rules for rounding off numerical values (*revised*).

1.1.1 This method applies to solid rubber of a hardness not less than 35 IRHD and to gases, such as air, nitrogen, oxygen, hydrogen, liquefied petroleum gas (in gaseous form) and coal gas. Errors may be introduced if the gas used, appreciately swells the rubber under test.

2. TERMINOLOGY

2.0 For the purposes of this standard, the following definition shall apply.

2.1 Permeability of Rubber to Gases — The rate of volume flow of gas under steady state conditions referred to standard temperature and pressure, between opposite faces of a unit cube of solid rubber, when subjected to unit pressure difference and controlled temperature.

3. OUTLINE OF THE METHOD

3.1 The cavity of a test cell, maintained at a constant temperature, is divided by a disc test piece into a high pressure and a low (atmospheric) pressure side. The high pressure side is connected to a constant pressure gas reservoir or is of such a volume that, once filled, it stays at a practically constant pressure. The gas permeates into the low pressure side, which is of a very low volume and connected to a capillary tube; this is provided for measuring the permeated volume, while keeping or restoring the same low pressure within this side. Absolute pressure for the high pressure side is maintained in the range of 0·3 to 1·5 MPa* and for low pressure side it is maintained at the prevailing atmospheric pressure.

3.1.1 For normal comparison of permeability of different rubber vulcanizates, the test temperature is a standard laboratory temperature (27 $\pm$ 1°C), but higher preferred temperature may be used where conditions are required to approximate to the service temperature of rubber products. When higher preferred temperatures are used, the capillary tube shall be brought to the test temperature and maintained at that temperature throughout the test.

4. APPARATUS

4.1 Test Cell

4.1.1 Test cell has means of clamping the test piece round its periphery in a gas-tight manner so as to expose one surface to gas under pressure. The other surface of the test piece shall be supported against the load due to the gas pressure so that no appreciable deformation takes place. For this reason the low pressure side of the test cell shall be filled with a rigid, very permeable packing piece which may consist of a disc of micro-porous material, such as microporous ebonite, discs of microporous sintered stainless steel or discs of fine wire gauze or filter paper which completely fill

*1 MPa = 1 N/mm² = approx 10 kgf/cm².

the cavity. A means of indicating gas pressure with an error of not more than one percent shall be connected to the high pressure side of the cell.

4.1.2 The volume of the high pressure side of the cell shall be at least 25 cm^3 to minimize the pressure loss due to diffusion during a test which may last several hours. The internal volume of the test cell on the low pressure (atmospheric) side of the test piece shall be kept to a minimum by the use of permeable packing as described above and by small diameter passages through a dismountable coupling and tubing to the capillary tube. The total free volume between test piece and zero mark shall not exceed 2 cm^3. Test cells shall be of metal construction with sufficient mass to assist temperature stability, and shall be provided with a drilled pocket to hold a suitable temperature measuring device.

4.2 Temperature Measuring Device — Accurate to $\pm$ 0·2°C.

4.3 Volume Measuring Device

4.3.1 It consists fundamentally of a capillary tube of known and uniform cross section over the length used for volume measurements. Suitable cross sections are 0·7 to 2 mm^2 with an accuracy of within 1 percent in uniformity. The capillary tube shall be graduated or a graduated scale shall be held close to it on the long straight portion. It shall be suitably filled with a non-volatile liquid which does not dissolve the gas [suitable liquids are di-(2 ethylhexyl) sebacate or tritolyl phosphate, coloured with Sudan red].

4.3.2 The capillary tube may be mounted on the cell layer or may be provided with a by-pass valve in order to start the volume measurements after a conditioning period. The capillary tube may be either horizontally or vertically mounted. In the first case, only a drop of liquid may be used as index of volume variations. In the second case, a vertically adjustable reservoir of liquid connected by a T-piece to the lowest portion of the capillary tube shall be provided to restore the low side pressure. A microscope or a cathetometer may be used to observe the liquid position.

4.4 Arrangement for Maintaining Constant Temperature — Constant temperature bath or other means capable of maintaining the test cell at the required test temperature to within $\pm$ 1°C. The wall of the bath shall be arranged so that the outlet from the test cell will project through the side leaving the capillary dismountable coupling accessible.

Examples of suitable apparatus are shown in Fig. 1 and 2.

5. TEST PIECE

5.1 Shapes and Dimensions — The test piece shall be a disc of uniform thickness and of dimensions to suit those of the test cell, and may be either moulded or cut from a test sheet or a product. It is preferable to use a

moulded disc having on each face a circumferential rib or bead to fit into corresponding grooves in the clamping members. Where the test piece is a flat sheet, suitable 'C' rings shall be used to fit into grooves in the test cell. The overall variation (excluding beads) in thickness shall not exceed 10 percent of the mean thickness.

5.1.1 Suitable dimensions are 50 to 155 mm diameter with a free testing surface of 8 to 70 cm^2 thickness may be between 0·25 and 3 mm, the smallest thickness being advantageous for rubbers of low permeability, such as *iso*butene-isoprene rubber. Imperfections and pinholes shall be absent.

5.2 Number of Test Pieces — Two test pieces of each rubber shall be tested.

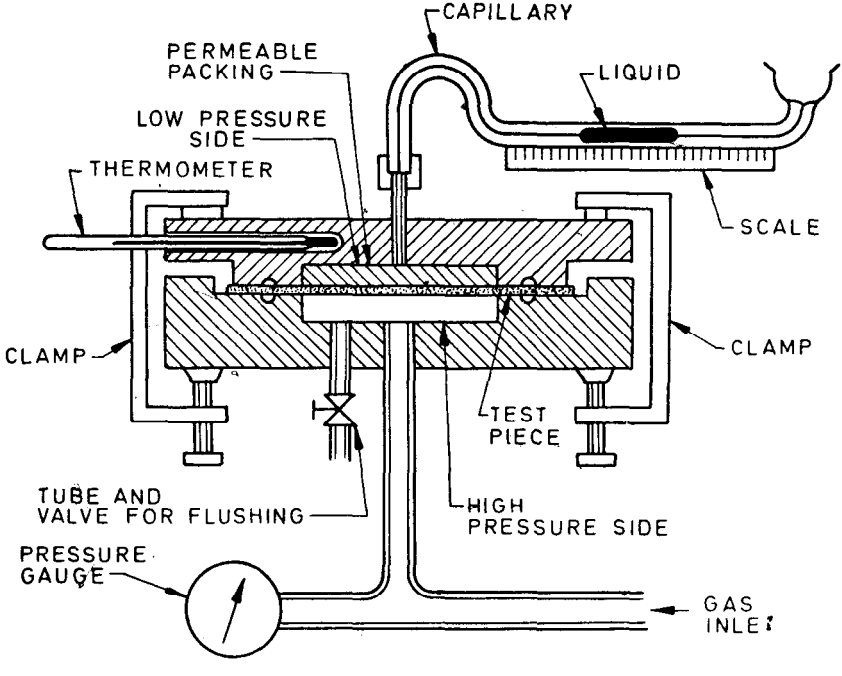

FIG. 1 APPARATUS WITH HORIZONTAL MEASURING DEVICE

6. TIME LAPSE BETWEEN VULCANIZATION AND TESTING

6.0 Unless otherwise specified for technical reasons the following requirements for time lapses shall be observed.

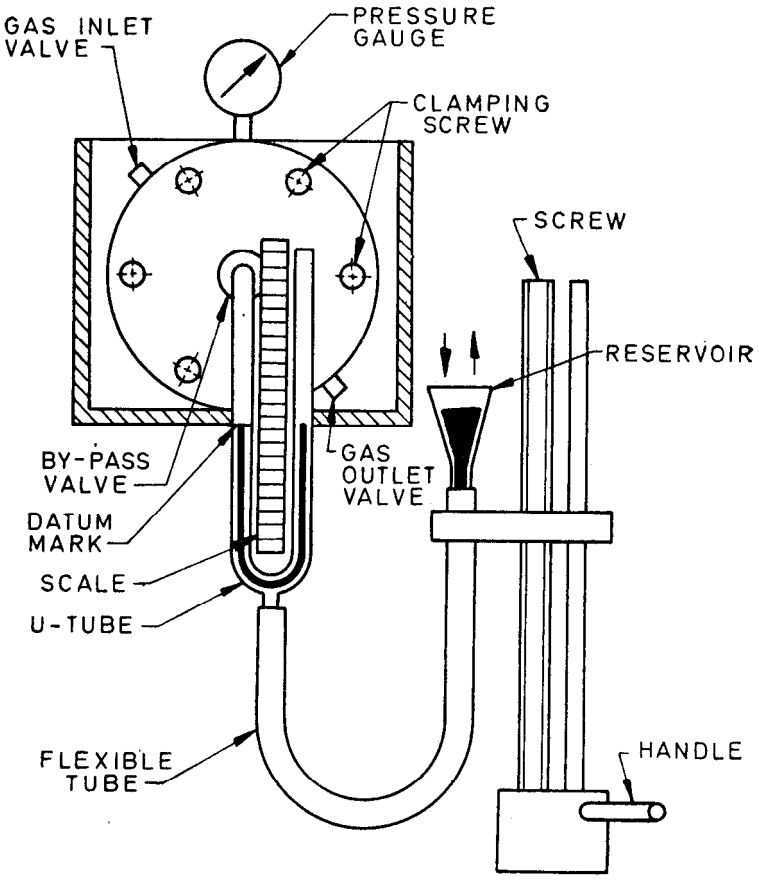

FIG. 2 APPARATUS WITH VERTICAL MEASURING DEVICE

6.1 For all test purposes the minimum time between vulcanization and testing shall be 16 hours.

6.2 For non-product tests the maximum time between vulcanization and testing shall be 4 weeks and for evaluation intended to be comparable, the tests as far as possible, should be carried out after the same time interval.

6.3 For product tests, whenever possible the time between vulcanization and testing shall not exceed 3 months. In other cases tests shall be made within 2 months of the date of receipt by the customer of the product.

7. TEMPERATURE OF TEST

7.1 For normal comparisons of permeability of different rubber vulcanizates, the test temperature shall be 27 ± 1°C. Higher temperatures may be used where conditions are required to approximate to service temperatures of rubber products. Such higher temperatures shall be selected from the list of preferred temperatures, namely:

40, 55, 70, 85, 100, 125, 150, 175, 200, 225 and 250°C

In any given test or series of tests intended to be comparable, the tolerance on the temperature shall be ± 1°C for temperatures up to 175°C and it shall be ± 2°C for temperatures of 200°C and above.

8. PROCEDURE

8.1 Preparation of Test Piece

8.1.1 Examine the test piece carefully for pinholes or imperfections within the area of the internal diameter of the cell (which is the effective test area). Care should be taken to avoid contamination of the surface between manufacture and test. Determine the thickness of the test piece in the test area as the average of six measurements each made to an accuracy of 0·02 mm. Insert the permeable packing in the shallow cavity behind the test piece on the low pressure side. Clamp the test piece securely round its periphery.

NOTE — It is permitted to use a minimum of vacuum grease on the clamping surface to secure gas-tightness. No grease shall be allowed to appear on the central area of the test piece.

8.1.2 Place the test cell in the constant temperature bath and connect to the test gas reservoir at the required pressure of test (0·3 to 1·5 MPa) depending on the permeability of the test piece. Maintain the pressure of gas to within ± 2·5 percent during the test.

8.2 Conditioning of Test Piece — The assembled apparatus shall be allowed to remain at the test temperature for minimum of 16 h, or where the approximate value of the diffusivity is known, for a minimum time t, in seconds, derived from the following equation:

$$t = \frac{b^2}{2Q} \times S = \frac{b^2}{2D}$$

where

b = thickness of the test piece in metres;

Q = permeability coefficient in square metres per pascal second [$m^2/(Pa.s)$];

D = diffusion coefficient in metres squared per second; and

S = solubility constant (volume of the gas dissolved per unit volume of the test piece at unit pressure) in reciprocal pascals Pa^{-1}.

This minimum time t ensures that the diffusion of gas through the test piece, and hence the gas flow through the test piece, may reach the steady state corresponding to the right-hand (straight) portion of the curve in Fig. 3. The left-hand portion of this curve indicates the initial approach to steady conditions due to diffusion through the test piece. The strictly linear portion of the curve only is used for permeability measurement.

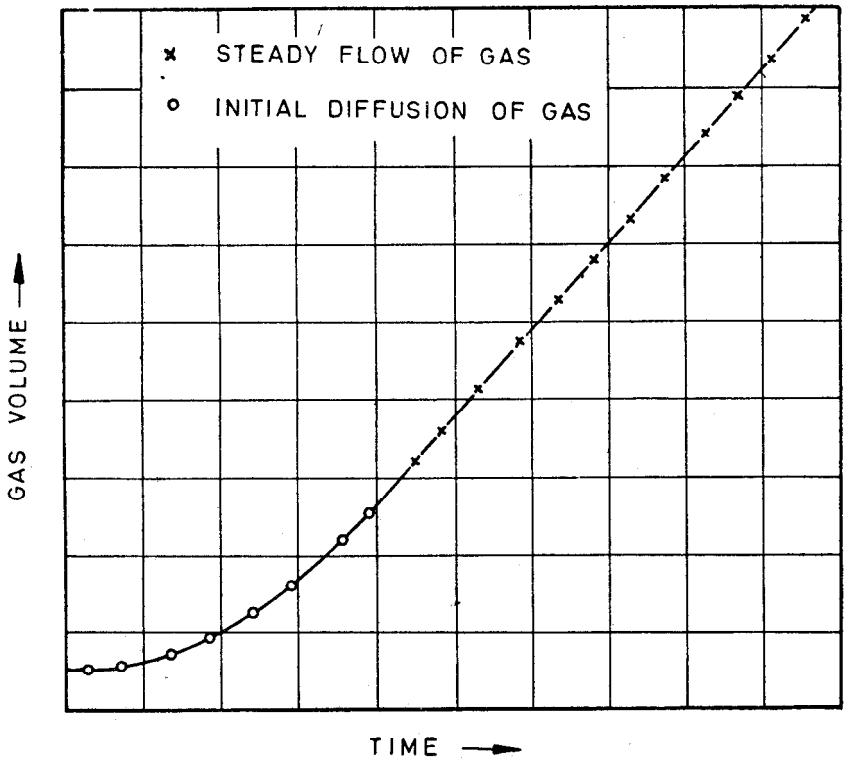

FIG. 3 TYPICAL TIME/GAS VOLUME CURVE

8.3 Determination of Flow Rate

8.3.1 Charge the capillary tube, calibrated as described in **8.4**, with the non-volatile liquid specified in **4.3.1**. Mount on the test cell and close the by-pass valve, if any. As the gas diffuses through the test piece, the meniscus is displaced. If the capillary tube is mounted vertically as in the apparatus shown in Fig. 2, compensate the levels in both legs of the U-tubes by level modification of the reservoir as often as possible, always directly before reading. Only the levels in the U-tube are to be considered, because the level in the compensator cannot agree with the other levels due to the capillary effect of the U-tube.

8.3.2 Start a stop-watch when the meniscus crosses the zero mark (zero time).

8.3.3 Record readings of the position of the liquid in the capillary tube at intervals of about 2 minutes throughout the test, which typically has a duration of 10 to 60 minutes after steady state.

8.3.4 Trace a time-length diagram; it must not show any appreciable departure from linearity after reaching a steady state. If this occurs, the test result shall be rejected and the test repeated.

8.4 Calibration of Capillary Tube — Introduce into the capillary tube a quantity of mercury to fill a length of about 20 mm (0·2 to 0·6 g). Keeping the tube horizontally mounted, perform the measurement of the length L of the mercury in several positions over the length of the tube (five to ten positions), with an accuracy of 0·02 mm. Weigh the capillary tube with and without the mercury, to the nearest milligram. The cross section, A_1 in millimetres squared, of the capillary tube in each position in given by:

$$A_1 = \frac{m_1 - m_2}{L.\rho}$$

where

m_1 = mass in mg of the capillary tube with mercury;
m_2 = mass in mg of the capillary tube without mercury;
L = length in mmHg in the capillary tube; and
ρ = density in g/ml of mercury.

The differences between any of the measurements of the cross section A_1 shall not exceed 1 percent of the mean value.

9. EXPRESSION OF RESULTS

9.0 The permeability shall be calculated in the following manner.

9.1 Pressure on the low pressure side shall be deduced from a barometer reading using the following relation:

1 mm of mercury at 0°C = 133·322 Pa

This may be corrected for thermal expansion of the mercury and the scale, but no sea-level reduction shall be applied.

9.2 Pressure on the high pressure side shall be deduced from the manometer reading (if this is relative, P_1, should be added).

9.3 Co-ordinates of one pair of points (or averages from many pairs) of the traced time-length diagram give corresponding $\triangle t$ and $\triangle L$ values.

9.4 Permeated volume, reduced to gas reference conditions, is:

$$\triangle V = A_1 \triangle L \frac{273\ p_1}{101\ 300\ T} = \frac{0{\cdot}002\ 7\ A_1\ \triangle L\ p_1}{T}$$

9.5 Permeability may be calculated as follows:

$$Q = \frac{b\ \triangle V}{\triangle t A_2 (p_2 - p_1)} = \frac{0{\cdot}002\ 7\ b\ A_1\ \triangle L\ p_1}{\triangle t\ A_2\ T\ (p_2 - p_1)}$$

where

A_1 = cross section of the capillary tube, in sqaure metres;

A_2 = free area of the test piece, in square metres;

b = thickness of the test piece, in metres;

$\triangle L$ = change in length of displacement of meniscus, in metres, obtained on the linear part of the curve, during time interval of $\triangle t$ seconds;

p_1 = pressure (absolute) on the low pressure side, in pascals;

p_2 = pressure (absolute) on the high pressure side, in pascals;

Q = rubber permeability, in metres squared per pascal second [m^2/(Pa.s)];

T = test temperature (absolute), in kelvin;

$\triangle t$ = time interval in seconds, for a given change in length of displacement of the meniscus;

$\triangle V$ = permeated volume of gas, at reference conditions, in cubic metres, during the time interval of $\triangle t$ seconds;

273 = gas reference temperature (absolute), in kelvin; and

101 300 = gas reference pressure (absolute), pascal.

For each set of two test pieces, the two values of permeability must be within 10 percent of their mean. If they are not, determine the permeability on a further set of two test pieces and calculate the mean value of the four test results.

10. TEST REPORT

10.1 The test report shall include the following particulars:

a) Sample details:
   i) a full description of the sample and its origin;
   ii) compound details, cure time and temperature, where appropriate; and
   iii) method of preparation of test pieces from sample, namely, whether moulded or cut and their dimensions.

b) Test details:
   i) the temperature of test;
   ii) the gas used in the test; and
   iii) the gas pressure on the high pressure side.

c) Test results:
   i) number of test pieces tested; and
   ii) the mean value of the permeability.

d) Date of test.

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